THE OPEN UNIVERSITY OF SRI LANKA

B.Sc. Degree Programme

Level 5 –Assignment Test II – 2013 / 2014

CMU 3128 – INSTRUMENTAL METHODS IN CHEMICAL ANALYSIS

Duration: One hour

Date and time: 26th September, 2014

Time: 9.30 a.m. - 10.30 a.m.

Reg. No.....

Question number	marks
1	
2	
Total	

Instructions to students

Answer all questions in the spaces given. Additional sheets will not be marked.

1. (A) Briefly explain the following related to radiochemical methods.

(i) Liquid scintillation detectors (mechanism and advantages)

(ii) K electron capture (how it happens and changes taking place)

(20 marks)

(B) (i) What is the **mechanism** behind Gas Ionization detectors?

(ii) Give **one advantage** and **one disadvantage** of Geiger Muller Counter.

(iii) What are the modifications introduced in the Gas Flow counter in order to overcome the disadvantages in the Geiger Muller Counter?

(15 marks)

(C) Briefly describe how **neutron activation analysis** is used in **quantitative analysis.**

(10 marks)

(D) To a sample having Iodine, 10 mg of $^{131}_{53}I$ with an activity of 1600 cpm was added. After mixing, 40 mg of Iodine having an activity 800 cpm was purified and separated. What is the weight of iodine in the original sample?

(10 marks)

2. (A) **Give reasons** for the following statements.

(i) Electrophoresis is not a chromatographic method.

(ii) The affinity of Ca^{2+} is greater than that of Mg^{2+} for a cation exchange column.

(20 marks)

(B) A column filled with silica was run with acetonitrile to separate compounds A and B in a mixture. The retention factors for A and B were 5 and 4.8 respectively.

(i) Name the **principle** behind separation here.

(ii) Comment **very briefly** on the separation of the compounds A and B using the above information.

(iii) Calculate the **selectivity factor** for A and B.

(iv) What is more **polar,** A or B? **Give reasons** for your answer.

(v) Suggest a method to **improve selectivity** of A and B using the same column.

(25 marks)

Name:.....

Address:.....

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CMU 3128 – INSTRUMENTAL METHODS IN CHEMICAL ANALYSIS - ANSWER GUIDE

1. (A)

(i)Used to detect low energy β particles; Here the sample is mixed with a solvent (liquid) & then with a scintillator. A scintillator (or a scintillator compound) is a compound capable of fluorescing. The radiation emitted by the sample is first absorbed by the molecules of the solvent and then by the scintillator resulting fluorescence. Advantages of these detectors are the short dead time & the ability to detect low energy β particles.

(ii)It is a made of decaying specially in isotopes where n:p ratio is low. When an electron in the K shell is attracted by a proton in the nucleus, an electron in an outer shell comes to fill the vacant K shell emitting X rays. This is called K electron capture. Here with the combination of the electron and the proton in the nucleus, a neutron is resulted. Therefore, the atomic number is decreased by 1 and there is no change in the mass number

(B)

(i) Radiation is passed through an inert gas which absorbs the radiation and form positive ions of the gas releasing electron. The cations and electrons are allowed to go through an electric field and as a result, there will be a change in current which is measured.

(ii) Advantages: 1) Simple 2) Inexpensive

Disadvantages: 1) Only high energy β particles & low energy γ & x rays can be determined. (or α & slow β particles cannot be determined)

2) High energy γ particles might not result a count.

3) Cannot distinguish between radiations of different energy.

(iii) The sample is introduced to the gas chamber directly.

(C)The element must have a naturally occurring isotope that can react with a neutron. By capturing the neutron, an unstable isotope is resulted with a moderate half life (not too short or too long). This unstable isotope emits γ rays or β rays without any interference which is characteristic. The activities of these rays are proportional to the amount of the naturally occurring isotope.

(D) R_s = 800 cpm R_t = 1600 cpm m_t = 10 mg m_s = 40 mg

$$\frac{Rt}{Rs} = \frac{mt+mx}{ms} \qquad m_x = \frac{Rt}{Rs} m_s - m_t = \left(\frac{1600}{800} \times 40\right) = 80 - 10 = \underline{70\ mg}$$

2.(A) (i) In chromatographic methods, there are two phases- Stationary phase & mobile phase. In electrophoresis, there is no mobile phase.

(ii)Ca^{2+} & Mg^{2+} - Same charge but the size of hydrated Mg^{2+} is more than that of Ca^{2+} (or size of Mg^{2+} is less than Ca^{2+} & Ca^{2+} is having a smaller hydration sheath) Therefore, Affinity of Ca^{2+} is more than Mg^{2+}.

(B) (i) Adsorption

(ii) Separation is poor.

(iii) Selectivity factor ($\propto$) = $\frac{Retention\ Factor\ of\ A}{Retection\ Factor\ of\ B} = \frac{5}{4.8} = 1.04$

OR Selectivity factor ($\propto$) = $\frac{Retention\ Factor\ of\ B}{Retection\ Factor\ of\ A} = \frac{4.8}{5} = 0.96$

(iv) A is more polar than B.

Silica is more polar than that of acetonitrile. According to the retention factors of A & B, A is retained more in the column than B. Therefore, A should be more polar than B.

(v) Make the mobile phase less polar than acetonitrile. OR Change the stationary phase.